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Settling Test Using Simulants to Evaluate Uranium Metal Distribution in K Basin Sludge

A. J. Schmidt
M. R. Elmore

April 2002



Prepared for the U.S. Department of Energy
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Spent Nuclear Fuel Project
Sludge Handling Group

Pacific Northwest National Laboratory
Richland, Washington 99352

Summary and Conclusions

This document presents the results of a large-scale settling test conducted with a K Basin sludge simulant that included metallic tungsten/cobalt (W/Co) fragments (density $\sim 14.5 \text{ g/cm}^3$) as a surrogate for uranium metal (density 19 g/cm^3). The objective of the testing was to gain insight into how uranium metal is likely to be distributed within the K Basin sludge loaded into the large-diameter containers (LDCs) that will be used for storage at T Plant. In the LDCs, uranium metal will react with water and generate heat and hydrogen gas. During loading, transportation, and storage operations, the uranium metal distribution in the LDCs will have an impact on the thermal stability. A settling model^(a) has been developed to predict the uranium metal distribution during loading to generate input conditions for modeling the thermal stability of the sludge in the LDCs.^(b) The results from the settling test discussed here will be compared against predictions developed from the settling model. This settling test was conducted by Pacific Northwest National Laboratory under contract to the Fluor Hanford Spent Nuclear Fuel (SNF) Project Sludge Handling Group.

A five-component simulant was formulated for the settling test to approximate the average particle size distribution and particle density of a 40/60 volume basis mixture of K East Basin canister and floor sludge. For the test, 20 jars (batches) were prepared, containing 1 liter of sludge simulant each. A clear acrylic column, 1 ft in diameter (OD) and 5 ft tall, was used as the settling vessel. To simulate flow to the LDC, supernatant in the upper part of the settling column was recirculated using a peristaltic pump. Water was removed 6 in. from the top of column and discharged back into the column at about 1.2 gpm [discharge point: center of column, 9 in. below the top of the column]. Before the test was started, three closed-bottom glass cylinders (2.5 in. OD x 12 in. high) were placed on the bottom of the column to collect core samples of the settled sludge.

For the test, a batch of sludge (1 L) was added to the top of the column (uniformly over the cross section of the column) once every 20 minutes. The sludge settled through 4 to 5 ft of water/slurry before accumulating on the bottom of the column. Twenty minutes after the last batch addition, the recirculation loop was turned off, and the suspended particulate was allowed to settle. The final settled sludge depth in the column was about 11.5 in.

About 40 hours after the last sludge addition, clarified supernatant was removed and the lower portion of the column (containing the settled sludge) was transported (about 3 miles) via a pickup truck for X-ray nondestructive evaluation (NDE) analysis. Because X-rays could not penetrate the dense W/Co, the distribution of W/Co could be examined within the settled sludge matrix. However, the X-rays also could not penetrate the entire column thickness; consequently, images were captured by focusing the X-ray about 2 to 3 in. in from the edge of column. The column was rotated on a turntable to provide 360 degree imaging. Once the imaging of the column was completed, the closed-bottom sample cylinders were removed from the sludge column, and X-rays taken.

(a) Plys, M. G. 2002. "Sludge Settling & Segregation During Loading." Letter Report FAI/02-08 from Fauske and Associates, Inc., to Hanford Spent Nuclear Fuel Project, Fluor Hanford.

(b) Heard, F. J. 2002. "Safety Basis Thermal Analysis of the Sludge Transport System." Correspondence number FH-0200768, transmitted in a letter to R. M. Crawford (FFS) from M. J. Schliebe (FH) on March 18, 2002. [A final report that includes the settling model is nearing publication; expected document number SNF-9955.]

Key observations and results from the settling test and the subsequent NDE are summarized below:

Settling Model Verification

- Twenty well-demarcated sludge layers were formed in the settled sludge. Each layer corresponded to the addition of one batch of sludge simulant. Within these primary layers, sub-layers, enriched in one or more of the simulant components were visible. The primary layers were about ½ in. thick, with the exception of the top layer. The top layer was about 1.1 in. thick as a result of the additional particulate that settled after the recirculation pump was shut off. These settling results are in accord with the overall character predicted by the settling model. The settling model predicts layer formation when sludge is added to the LDC in serial additions, where each added batch segregates into sub-layers enriched in and lacking in uranium metal.
- Based on observations of the layers, the overall settled sludge depth, and the manner in which the sludge additions were made, the upper 10% to 15% of the settled sludge bed was expected to contain little or no W/Co fragments. The absence of W/Co in the top portion of the sludge bed results from the final layer that formed when the fine particulate settled after the pump shut off, and because W/Co in the twentieth (final) batch was mostly captured in the sludge layer created by the sludge from the nineteenth batch. The settling model also predicts the existence of a top layer, formed by longer-term settling, that contains no metal.
- During the careful movement of the column to the transport vehicle and the subsequent transport to the NDE facility, accelerations/vibrations caused the settled sludge to liquefy, and the visually well-defined layers partially collapsed. Results from X-ray analysis of the settling column (after transportation/liquefaction) showed depletion of W/Co in the top ~35% of the column vs. the expected 10% to 15% depletion. In the bottom 65% of the column, uniform distribution of W/Co was observed. No evidence of W/Co segregation to the very bottom of the column was found.
- After NDE was completed on the column, the three 2.5-in.-diameter sample cylinders were removed. Twenty reasonably well-defined layers of sludge were observed in each sample cylinder (i.e., no evidence of liquefaction, but some compaction).
- X-rays showed the vertical distribution of the W/Co fragments in the 2.5-in.-diameter sample cylinders was very uniform, and no evidence of gross W/Co segregation was found. The concentration of W/Co fragments in the upper 5% to 15% of the sample cylinders appeared to be reduced. From these results, it can be inferred that before transportation (and liquefaction) the distribution of W/Co in the settling column was essentially uniform in the lower 85% to 90% of the column.

Related Observations on Settled Sludge Behavior During Handling

- As a result of sludge reconsolidation after liquefaction during transport, the settled sludge depth in the settling column decreased about 15%, from ~11.5 in. to 9.9 in. Before transport, only a thin layer of water remained above the settled sludge. After transport, 1.3 in. of clarified supernatant was observed above the sludge. No changes were observed in the sludge depth in the 2.5-in.-diameter sample cylinders.

- After the 2.5-in.-diameter sample cylinders were removed, X-rays were again taken of the settling column. Even though the settled sludge was significantly disturbed by withdrawing the cores, the distribution of the W/Co was largely unaffected (i.e., no significant segregation as the sludge and water moved to fill in the voids left behind when the cylinders were pulled).

Conclusions

- The settling model proposed for the LDC sludge filling operation was clearly confirmed by the settling test. Because of the methods used and consistent behavior observed, no further settling model confirmation testing is proposed.
- The observed instability of the defined layers in the 1-ft-diameter settling column suggests that some liquefaction could occur in the 5-ft-diameter LDCs. However, the settling test was not designed to simulate LDC transport nor to evaluate the effects of liquefaction; consequently, it is speculative to extrapolate the test observations to the LDC at this time. Because of the relative ease at which liquefaction occurred in the 1-ft-diameter settling column, further evaluation of the liquefaction potential of the LDC is warranted.

Acknowledgments

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1.0 Introduction

Two water-filled concrete pools [K East (KE) and K West (KW) Basins] in the 100K Area of the Hanford Site originally contained more than 2100 metric tons of N Reactor fuel elements stored in aluminum or stainless steel canisters. The fuel is currently being retrieved and processed for dry storage. During the time the fuel had been stored in the K Basins, approximately 52 m³ of heterogeneous solid material (sludge) accumulated in the canisters, as well as on the floor and in the associated pits. This sludge consists of various proportions of fuel; structural corrosion products; windblown material; and miscellaneous constituents, such as ion exchange material (both organic and inorganic) and paint chips (Makenas et al. 1996-99). Pearce (2001) describes the inventory and compositions of all K Basin sludge materials in detail.

Management plans for the spent nuclear fuel (SNF) call for the sludge to be packaged, shipped, and stored at T Plant in the Hanford 200 Area until final processing at a future date. The KE Basin sludge will be retrieved and stored in large-diameter containers (LDCs) that are approximately 5 ft in diameter and 10 ft high.

K Basin sludge contains metallic uranium and uranium oxides that will react with water to corrode, which in turn generates heat and hydrogen gas. To meet the Fluor Hanford SNF Project objectives, the sludge must be retrieved, staged, transported, and stored in systems designed to provide a rate of heat removal that prevents the water in the sludge from reaching boiling temperatures. The spatial distribution of uranium metal fuel particles in the sludge is a parameter that must be known or estimated to quantify the energy density and the rate of heat removal for the sludge contained in the LDCs.

A model^(a) recently developed for the Fluor Hanford SNF Project predicts particle settling during loading of LDCs. This model predicts that a relatively uniform distribution of metallic uranium fuel particles occurs when multiple layers are loaded sequentially into the LDC. The output from the settling model is being used as input to the LDC thermal stability model.^(b)

This report documents the results of a large-scale settling test conducted by Pacific Northwest National Laboratory (PNNL) under contract to the Fluor Hanford SNF Project, Sludge Handling Group. The objective of the testing was to gain insight into how uranium metal is likely to be distributed within the K Basin sludge loaded into the LDCs.

For the settling test, a K Basin sludge simulant was used that included metallic tungsten/cobalt (W/Co) fragments (density ~14.5 g/cm³) as a surrogate for uranium metal (density 19 g/cm³). Twenty 1-L batches of the K Basin sludge simulant were sequentially added to a 1-ft-diameter by 5-ft-high clear acrylic settling column. During the test, supernatant was recirculated in the upper portion of the column to simulate general flow patterns associated with sludge addition to the LDCs. After all batches of sludge were added, the sludge was allowed to settle and consolidate for about 40 hours. Clarified supernatant

(a) Plys, M. G. 2002. "Sludge Settling & Segregation During Loading." Letter Report FAI/02-08 from Fauske and Associates, Inc., to Hanford Spent Nuclear Fuel Project, Fluor Hanford.

(b) Heard, F. J. 2002. "Safety Basis Thermal Analysis of the Sludge Transport System." Correspondence number FH-0200768, transmitted in a letter to R. M. Crawford (FFS) from M. J. Schliebe (FH) on March 18, 2002. [A final report that includes the settling model is nearing publication; expected document number SNF-9955.]

was removed, and the settled sludge was transported approximately 3 miles to a nondestructive examination (NDE) facility where the W/Co distribution as a function of sludge depth was determined by X-ray. Accelerations and vibrations associated with transporting the settled sludge to the NDE facility caused the settled sludge to liquefy.

The results from this settling test will be compared against predictions developed from the settling model. Section 2.0 describes the settling test equipment, the simulant, and the test parameters and procedures. Test results and observations are presented in Section 3.0.

2.0 Test Equipment, Materials, and Parameters

This section describes the test equipment; the basis for selecting the simulant composition, along with its physical properties; and the parameters and procedures used in the testing. The settling test was conducted on February 26, 2002, at the PNNL Process Development Laboratory East (PDLE) high bay facility located in North Richland. The NDE analyses were performed by Cogema in the 306E Building located in the Hanford 300 Area.

2.1 Settling Test Equipment

The equipment used for the settling test mainly consisted of a settling column, a peristaltic pump, glass sample cylinders, and an X-ray realtime unit. The test setup sought only to simulate as closely as possible the general settling behavior of the sludge simulant under nominal "LDC-like" conditions. A fully representative model of the LDC and its internals (i.e., filter area, inlet, inlet flow deflector, etc.) was beyond the scope of this testing.

2.1.1 Settling Column

Figure 1 shows the settling column and pump; Figure 2 is a photograph of the settling equipment. The settling column is constructed of clear acrylic and is 5 ft high, with a 1-ft outside diameter (11.5-in. ID). The column has a flanged joint 14 in. from the bottom to provide easy access to settled sludge. The column also includes a penetration 6 in. below the top for water recirculation. During the test, the water level was maintained about 3 in. below the top of the column. While the LDC has a dished bottom, the settling column has a flat bottom. If a dished bottom had been used in the settling test, the W/Co distribution for the first several batches of sludge simulant may have been different.

2.1.2 Pump

A peristaltic pump was used to recirculate water/slurry in the upper portion of the column to simulate the once-through flow in the LDC. The recirculation rate was maintained at an average of 1.2 gpm during the settling test. The slurry was discharged through a 7.5-mm (0.295-in.)-ID tube into the approximate center of the column, about 9 in. below the top of the column.

2.1.3 Sample Cylinders

Three closed-bottom glass cylinders [2.5 in. OD (2.3 in. ID) x 12 in. high] were arranged in a straight line on the bottom of the column (Figure 3). One was located at the center of the settling column, one along the wall, and one halfway between the wall and center. The sample cylinders provided the flexibility to pull out cores of the settled sludge with minimal disturbance to the settling profile, and provided a contingency for detailed analyses of the cores if the X-rays could not penetrate through the entire thickness of the settling column.

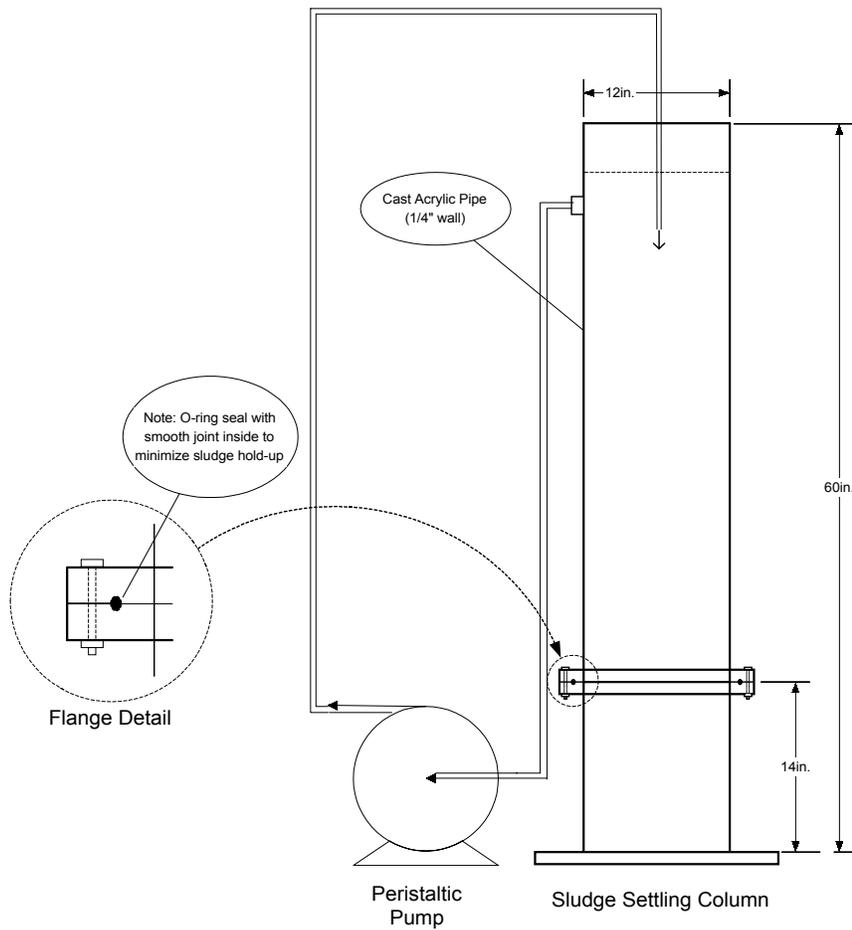


Figure 1. Diagram of Sludge Settling Equipment

2.1.4 NDE X-ray System

The X-ray realtime (RTR) unit used for this testing consists of a Philips 450kv X-ray machine, a 9-in. image intensifier, a Cohu low light video camera, a zoom lens, an Argus image processor, and a 13-in. Sony monitor. The sludge column was placed on a turntable/lift in line with the X-ray tube and image intensifier. The image intensifier converts the X-rays to visible light, and the camera sees the image of the sludge container at the image intensifier output. The video image is then sent to the image processor for improvement, and then to an SVHS video recorder and onto the video monitor. This X-ray system is similar to the RTR unit used to examine 55-gallon TRU waste drums at the Hanford Waste Reviewing and Packaging (WRAP) facility for the Waste Isolation Pilot Plant (WIPP).



Figure 2. Settling Column and Pump Set-up

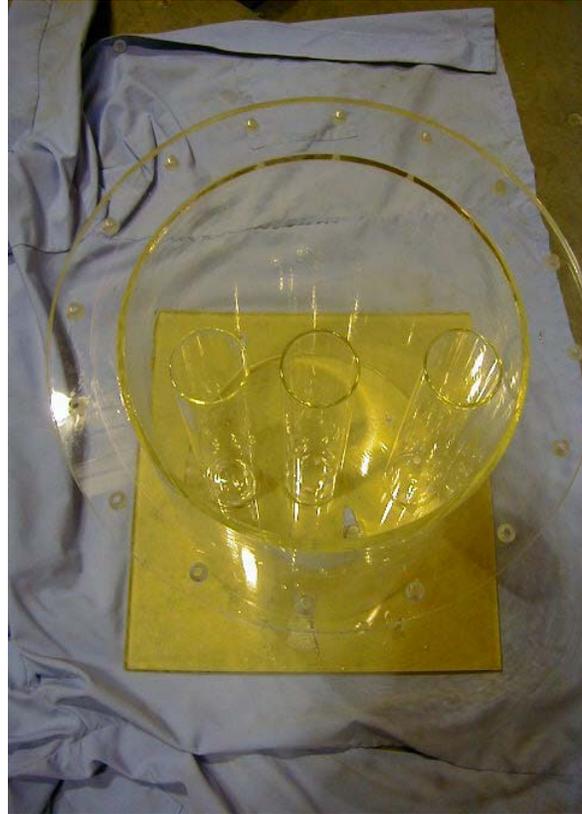


Figure 3. Arrangement of Sample Cylinders

2.2 K Basin Sludge Settling Simulant

The simulant developed for the settling test resembled a nominal 40/60 KE canister/floor sludge mixture. This simulant was based in part on the K Basin simulant developed for the LDC Proof-of-Principle (PoP) Test.^(a) On a dry weight basis, the LDC PoP simulant consisted of: 8 wt% tungsten/cobalt (W/Co) metallic fragments (250 to 6350 μm); 17 wt% flyash (typically between 0.5 and 180 μm); 37.5% Min-U-Sil-40 (a ground silica, with the majority of material between 5 and 20 μm); and 37.5% foundry sand (majority of particles between 100 and 400 μm). The particle size distribution and settling behavior of the LDC PoP simulant were found to be very similar to those of KE Basin floor sludge. The yield strength of the LDC PoP simulant was found to be 93 Pa after 24 hours, and 106 Pa after 48 hours.

The LDC PoP simulant does not contain a fine, high-density material to represent non-metallic uranium species, nor a constituent to represent the larger-diameter non-uranium material found in the K Basin sludge. Therefore, modifications were made to the LDC PoP simulant to create an improved simulant for the settling tests, as described below.

(a) Baker, R. B. and A. J. Schmidt. November 20, 2001. "Summary Recommendations for Sampling to Support Proof of Principle Testing of the Sludge Transport System Large Container." Letter to G. A. Sly, 01-SNF/RBB-006, Spent Nuclear Fuel Project, Fluor Hanford, Richland, WA.

2.2.1 W/Co to Simulate Metallic Uranium

The material used as a surrogate for uranium metal was KENFC272 ¼-in.-down (8.00% Co, max; 5.70% C, max; 0.20 wt% Ti, max; balance is W), referred to as W/Co. The particle density of W/Co is $\sim 14.5 \text{ g/cm}^3$, while the bulk dry density is $\sim 10 \text{ g/cm}^3$. Particles range from 250 μm to 6350 μm in the existing W/Co feedstock, with approximately 61 wt% made up of particles greater than 2000 μm , and only 0.54 wt% made up of particles less than 250 μm (2.32 wt% is less than 500 μm). It is expected that most of the uranium fragments in actual K Basin sludge will be between 500 and 2000 μm (Delegard et al. 2000; Pearce and Plys 2001). It is not expected that uranium metal particles in the 2000- to 6350- μm range would have a tendency to segregate any differently than those less than 2000 μm . Therefore, the W/Co feedstock was sieved to remove all fragments larger than 2000 μm (US sieve #10).

2.2.2 Stainless Steel Particles to Simulate Non-metallic Uranium

Uranium metal corrodes to form uranium oxides and hydrates with a range of particle densities between about 5 and 11 g/cm^3 . From an analysis developed by Schmidt and Delegard (2002), the average particle density of the non-metallic uranium species in KE floor and canister sludge was estimated to be 7.53 g/cm^3 . In KE canister sludge samples, significantly higher uranium concentrations were found in sample fractions containing particles less than 250 μm (Elmore 2000). Therefore, stainless steel powder, with a particle density of $\sim 7.8 \text{ g/cm}^3$, was determined to be a suitable surrogate for the non-metallic uranium. A low-cost stainless steel powder, in which all particles are less than 125 μm and 38% to 48% are less than 44 μm , was identified for the settling simulant. [Note: While the majority of the non-metallic uranium particles are less than 250 μm , non-metallic uranium particles/fragments/agglomerates larger than 250 μm are present in K Basin sludge samples. As an example, about 40 wt% (dry basis) of KE canister sludge sample 96-06 was made up of particles greater than 710 μm (Makenas et al. 1997). On a dry weight basis, the uranium content of 96-06 was about 83 wt%, which would indicate the larger-diameter particles contained an appreciable quantity of uranium. Therefore, the use of stainless steel powder does not simulate all sizes of non-metallic uranium species present in all cases of the actual sludge. No single sludge simulant could encompass all bounds associated with the various sludge types in the KE Basin. However, the simulant chosen is believed to be representative of the parameters of primary interest to settling.]

2.2.3 Fragments to Simulate Larger-Diameter Non-uranium Sludge Particles

On the average, approximately 25 wt% of the actual KE floor and canister sludge is made of particles greater than 500 μm . Limited data on particle density as a function of particle size is found in Bredt et al. (1999). For canister sludge, particles between 500 and 1410 μm exhibit a density of 4.63 g/cm^3 . The particle densities of larger canister sludge particles are 2.89 g/cm^3 (1410 to 4000 μm) and 2.23 g/cm^3 (4000 to 6350 μm). For floor sludge, the particle densities are 3.14 g/cm^3 (< 250 μm), 2.78 g/cm^3 (250 to 500 μm), and 2.63 g/cm^3 (500 to 1410 μm).

In comparison, the average particle density of gibbsite, goethite, and silica (non-uranium species found in K Basin sludge) is 3.1 g/cm^3 . Given that the particle density of W/Co is 76% (14.5/19) of the particle density of metallic uranium, a sand blasting product, Kleen Blast, with a particle density of 2.8 g/cm^3 , was identified as a suitable surrogate for the larger-diameter-particle, non-uranium species. Kleen Blast, which is sold as a 15 to 30 mesh material, is roughly composed of particles that are a homogeneous mixture of 45% silicone, 23% iron oxide, 19% calcium oxide, and 7% aluminum. [Note: The use of a

single component to simulate all large non-uranium particles is a simplification of the complex composition of the actual K Basin sludge.]

2.2.4 Material to Simulate Fine Particulate

Consistent with the LDC PoP simulant, flyash and Min-U-Sil 40 were used as fine particulate in the settling simulant. The particle density of flyash is ~ 2.2 to 2.5 g/cm^3 , and the particles are between 0.5 and $180 \text{ }\mu\text{m}$. Min-U-Sil 40 is a ground, high-purity silica powder with a particle density of 2.65 g/cm^3 and a mean particle diameter of $11 \text{ }\mu\text{m}$.

Although originally present in the LDC PoP simulant, foundry sand was not included in the settling simulant. Kleen Blast overlaps a portion of the foundry sand particle size distribution, and higher quantities of flyash and Min-U-Sil 40 were needed to provide a simulant that better represents the low-end of the actual K Basin sludge particle size distribution.

2.2.5 Final Simulant for Testing

The target KE Basin sludge being represented in the settling test was a 40/60 KE Basin canister/floor sludge mix. Based on Plys and Pearce (2001) and the LDC PoP simulant, the settling simulant was developed as described below. [An initial simulant was developed that exhibited a settled density of 2.6 g/cm^3 and a void fraction of 0.6 . These initial results (settled density and void fraction) were used to calculate the dry basis composition of the final simulant. The final simulant exhibited a somewhat lower settled density and void fraction.]

- W/Co fragments: At safety basis conditions, the canister sludge contains $0.125 \text{ g/cm}^3 U_{(\text{metal})}$, and the floor sludge contains $0.023 \text{ g/cm}^3 U_{(\text{metal})}$. Therefore, a 40/60 mix will contain $0.4 \times 0.125 + 0.6 \times 0.023 = 0.0638 \text{ g/cm}^3 U_{(\text{metal})}$. The settling simulant was expected to have a settled density of 2.6 g/cm^3 , and a void fraction of about 0.6 . Therefore, on a dry weight basis, to match bounding conditions, the W/Co will be added at about $0.0638/(2.6 - 0.6) = 3.2 \text{ wt}\%$.
- Stainless steel powder: At safety basis conditions, the canister sludge contains $1.5 \text{ g/cm}^3 U_{(\text{total})}$, and the floor sludge contains $0.15 \text{ g/cm}^3 U_{(\text{total})}$. Therefore, a 40/60 mix will contain $0.4 \times 1.5 + 0.6 \times 0.15 = 0.69 \text{ g/cm}^3 U_{(\text{total})}$. $U_{(\text{non-metal})} = U_{(\text{total})} - U_{(\text{metal})} = 0.69 - 0.0683 = 0.63 \text{ g/cm}^3 U_{(\text{non-metal})}$. The settling simulant was expected to have a settled density of 2.6 g/cm^3 , and a void fraction of about 0.6 . Therefore, on a dry weight basis, to match bounding conditions for $U_{(\text{non-metal})}$ the stainless steel powder will be added at about $0.63/(2.6 - 0.6) = 32 \text{ wt}\%$.
- Non-uranium, large-diameter fragments, Kleen Blast: Approximately $25 \text{ wt}\%$ of the canister and floor sludge particles are larger-diameter particles ($0.25 \times 2.6 \text{ g/cm}^3 = 0.65 \text{ g/cm}^3$). On a dry weight basis, the fraction of larger-diameter particles = $0.65/(2.6 - 0.6) = 32.5\%$. After subtracting the $\text{wt}\%$ W/Co (also larger-diameter particles), on a dry weight basis, the Kleen Blast will be added at $29 \text{ wt}\%$ ($32.5 - 3.2$).
- On a dry weight basis, W/Co + SS powder + Kleen Blast = $64 \text{ wt}\%$. The remaining 36% of the simulant will consist of equal mass percentages of the fine particulate components from the PoP simulant, flyash and Min-U-Sil 40:
Flyash = $36 \times 0.5 = 18 \text{ wt}\%$
Min-U-Sil 40 = $36 \times 0.5 = 18 \text{ wt}\%$.

Table 1 summarizes the final simulant composition and provides the component particle densities and component particle size distributions (PSDs). Figure 4 compares the PSD of the simulant to the average PSDs from actual KE floor and canister samples. The target settled density for the simulant (based on KE floor and canister sludge safety basis values) was 1.9 g/cm³. The measured settled density was 2.24 g/cm³, about 18% above this target.

2.3 Settling Test Parameters/Procedures

Test instruction PNNL TI-43262-T03^(a) describes each step and the data to be collected for the settling test. The test instruction was reviewed and approved by the Fluor Hanford SNF Sludge Handling Group and SNF Sludge Safety and Modeling Group. The key test parameters, rationales, and procedures are summarized here.

Table 1. Simulant Composition and Properties

| | | W/Co | SS Powder | Kleen Blast | Flyash | Min-U-Sil 40 | Simulant |
|--|------|-------------|-----------|-------------|-----------|--------------|----------|
| Wt Fraction, dry | | 3.2 | 32 | 29 | 18 | 18 | 100.2 |
| Particle Density, g/cm³ | | 13.9 – 14.5 | 7.8 | 2.6 – 2.8 | 2.2 – 2.5 | 2.65 | |
| Measured Wt Fraction, wet | | 2.5 | 25 | 22 | 14 | 14 | 77.5 |
| Particle Size Distribution, Percent Less Than | | | | | | | |
| US Mesh | µm | W/Co | SS Powder | Kleen Blast | Flyash | Min-U-Sil 40 | Simulant |
| 10 | 2000 | 100 | 100 | 99.78 | 100 | 100 | 99.9 |
| 18 | 1000 | 34.33 | 100 | 78.94 | 100 | 100 | 91.8 |
| 35 | 500 | 5.66 | 100 | 47.48 | 100 | 100 | 81.8 |
| 60 | 250 | 1.32 | 100 | 15.98 | 100 | 100 | 72.5 |
| 140 | 106 | .23 | 83.6 | 1.71 | 97.8 | 100 | 62.7 |
| | 100 | .21 | 80.7 | 1.52 | 97.4 | 100 | 61.7 |
| 270 | 53 | .13 | 39.5 | 0.1 | 88 | 98 | 46.1 |
| | 30 | .01 | 17.3 | 0.1 | 75 | 94 | 35.9 |
| | 20 | 0 | 7.8 | 0 | 65 | 83 | 29.1 |
| | 10 | 0 | 1.3 | 0 | 45.6 | 46 | 16.9 |
| | 5 | 0 | 0 | 0 | 30 | 26 | 10.1 |
| | 1 | 0 | 0 | 0 | 7.8 | 7 | 2.7 |
| Additional Simulant Properties | | | | | | | |
| Bulk, untapped dry density = 1.74 g/cm ³ | | | | | | | |
| Bulk, tapped dry density = 2.17 g/cm ³ | | | | | | | |
| Settled wet density = 2.24 g/cm ³ | | | | | | | |
| Void Fraction = 0.51 | | | | | | | |

(a) Schmidt, A. J. February 22, 2002. PNNL Test Instruction TI-43262-T03, “K Basin Sludge Simulant Settling.” Pacific Northwest National Laboratory, Richland, WA.

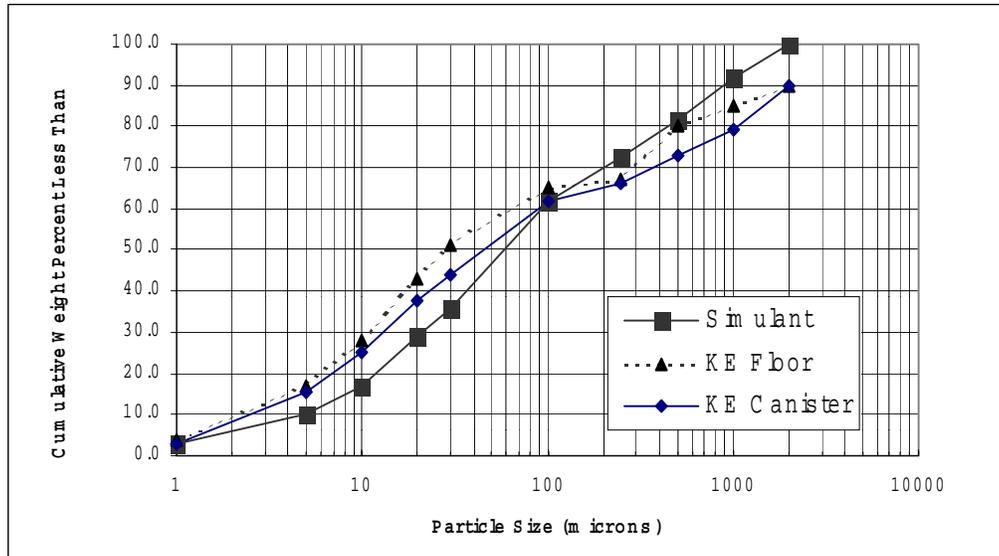


Figure 4. Comparison of the Particle Size Distributions of the Settling Simulant to Average of KE Floor and Canister Sludge Samples

2.3.1 Slurry Recirculation/Discharge Rate

During the settling test, the slurry recirculation rate averaged 1.2 gpm (4.5 Lpm). The active volume in the settling column was about 25.6 gallons; therefore, the effective average residence time in the settling column was about 21 minutes. In the LDC, the effective residence time will vary from about 12 to 35 minutes, depending on the inlet flow rate (30 to 90 gpm).

The pump discharge line into the settling column was submerged. The velocity at the point of discharge averaged approximately 5.6 ft/sec. The feed inlet to the LDC is currently a 1 ½ in. pipe. At a feed rate of 30 gpm, the discharge velocity will be 4.7 ft/sec. At 90 gpm, the feed discharge velocity in the LDC will be 14.2 ft/sec. Therefore, the discharge velocity in the settling test was in the lower range of what is expected in the LDC. For the settling test, no attempt was made to simulate the upper internals (i.e., inlet pipe discharge, flow deflector plates, filter array, etc.) of the LDC or the flow pattern other than very general circulation behavior. In the LDC, the upper slurry will be filtered and exhausted to the KE pool and will not be recirculated as was done in the settling test (for simplicity).

2.3.2 Sludge Simulant Addition Rate

At a recirculation rate of 4.5 Lpm, each 20-minute interval was equivalent to 90 L of feed slurry. Each liter of settled sludge simulant (2240 g) contained 1728 g of dry materials. Therefore, the effective total solids concentration in the feed for the settling test was 1.87 wt% total solids [100 x 1728 g / (90,000 g + 2240 g)].

2.3.3 Sludge Simulant Preparation and Addition Procedure

For batch-to-batch uniformity, the dry simulant components were individually weighed to prepare each batch of sludge. Water from the top of the settling column was added to each batch about 45 minutes before addition to the settling column. The simulant was well mixed and was tumbled on a jar roller for 10 to 20 minutes, then left undisturbed for 10 to 20 minutes to allow air bubbles to escape. The sludge simulant was then added to the column at carefully spaced 20-minute intervals. After the fourth addition, sludge was added by inverting the jar and sprinkling the contents through a 3/8-in. hole in the jar lid. Each addition took approximately 3 minutes (not including the time required to rinse the residual sludge simulant from each jar into the column).

2.3.4 Settling/Consolidation Time

The sludge was allowed to settle and consolidate for 40 hours before the settled sludge was moved to the NDE facility. Testing with the simulant showed that negligible consolidation occurred after 2 hours of settling time. In settling tests performed with actual K Basin sludge samples, very little sludge consolidation has been observed, in most cases, after 24 hours of settling (Bredt et al. 1999; Makenas et al. 1996, 1997).

3.0 Results and Observations

This section describes observations made during and after the settling test. Photographs taken during testing are provided to illustrate the settling behavior. The resulting W/Co fragment distribution shown in the X-ray images from NDE is also displayed here.

Before the sludge simulant was added to the settling column, the column was filled with tap water, and the recirculation pumping rate was set at about 1.2 gpm. With the peristaltic pump, a pulsed flow was observed. However, the pulse was not strong enough to vibrate the column or disturb the settled sludge. A small quantity of Min-U-Sil 40 was also added before starting the settling test to observe and videotape the circulation patterns in the column. The discharge flow influenced the circulation patterns to a depth of ~3 ft below the discharge point. In general the circulation patterns were gentle.

The first four sludge simulant additions were made using a combination of pouring and ladling sludge into the top of the column. Uniform distribution of the sludge components was difficult to achieve with these techniques. Consequently, subsequent additions entailed inverting the sludge jars and sprinkling the sludge into the column through a 3/8-in. hole in the lid. With this technique, a fairly uniform distribution of the sludge components was achieved across the column diameter.

Between the ninth and tenth sludge additions, the peristaltic pump was briefly shut off (< 30 seconds) and flow was reversed. This brief interruption in the column circulation patterns resulted in a thicker settled sludge layer between the ninth and tenth sludge layers in the column. After the last batch of sludge was added to the column, slurry recirculation was continued for 20 minutes. After the pump was shut off, the supernatant was largely clarified in 1 to 2 hours. The settled sludge depth was about 11.5 in. within 1 hour of the pump shutoff. After 40 hours of consolidation, the settled sludge depth was still about 11.5 in. Due to the inability to radially redistribute, the 12-in.-high sample cylinders were heaping full after the settling test, and the sludge level appeared to be ~0.5 in. higher in the cylinders than in the column (Figure 5a).

During the course of the test, the sludge settled in well-demarcated primary layers. The Kleen Blast, a black glassy material (29 wt% of the simulant on a dry basis), created a black band in the column for each batch of sludge added (Figure 5b). In these primary layers, sub-layers, enriched in one or more of the simulant components were formed. At the conclusion of the test, 20 well-defined layers were observed (Figure 5b), with the top layer being the thickest. The top layer included the fine particulate that did not settle until the recirculation pump was turned off. The overall settling behavior observed in the test is consistent with the overall character predicted by the settling model.^(a) The settling model predicts layer formation when sludge is added to the LDC in serial additions, where each added batch segregates into sub-layers enriched in and lacking in uranium metal.

Based on observations of the layers, the overall settled sludge depth, and the manner in which the sludge additions were made, the upper 10% to 15% of the settled sludge bed was expected to contain little or no W/Co fragments. The absence of W/Co in the top portion of the sludge bed results from the final layer that formed when the fine particulate settled after the pump shut off, and because W/Co in the twentieth (final) batch is mostly captured in the sludge layer created by the sludge from the nineteenth batch. The

(a) Plys, M. G. 2002. "Sludge Settling & Segregation During Loading." Letter Report FAI/02-08 from Fauske and Associates, Inc., to Hanford Spent Nuclear Fuel Project, Fluor Hanford.

settling model also predicts the existence of a top layer, formed by longer-term settling, that contains no metal.

After 40 hours of consolidation time, the clarified supernatant was removed. As designed, the lower portion of the column was unbolted and disassembled. The lower 14 in. of the column was placed on a dolly, rolled out to a transport vehicle (pickup truck), and transported ~3 miles (~10 minutes of driving time) from PDLE (North Richland) to the NDE facility (306E, 300 Area).

During the transport, vibrations caused the visually well-defined layers to partially collapse (i.e., liquefaction). The collapse of the layers was initiated by the mild agitation resulting from rolling the settling column to the pickup truck. Although a gentle route (well-maintained blacktop road surface) was taken, the layers were further disrupted during the drive to 306E. As a result of the liquefaction, the sludge volume decreased by about 15%, resulting in a sludge depth of 9.9 in. as the sludge consolidated and stabilized. Figure 6 shows the sludge column after transport to the NDE facility. The locations of the lead numbers used to demarcate the vertical position of the column during X-ray are shown in Figure 6a.

The settling column, as shown in Figure 6 (i.e., after liquefaction), was examined by X-ray. The W/Co fragments show up as dark particles in the X-ray views. The W/Co exhibits much greater X-ray stopping power than the stainless steel powder. The Kleen Blast, Min-U-Sil 40, and flyash are mostly transparent to the X-rays. The X-ray examination showed depletion of W/Co in the top ~35% of the column vs. the expected 10% to 15% depletion before liquefaction. In the bottom 65% of the column, very uniform distribution of W/Co was observed. Furthermore, no significant concentrations of W/Co fragments were observed resting on the very bottom of the column. Figure 7 shows X-ray images at various positions in the settling column. While the distribution of W/Co was relatively clear when viewing the videotape, the resolution of the captured images in Figure 7 is marginal. In Figure 7a (top of sludge column), a sample cylinder (2.5 in. diameter) is visible and W/Co fragments can be seen inside the cylinder. However, the adjacent area surrounding the cylinder is free of W/Co fragments. Figures 7c through 7f show the reasonably good W/Co distribution in the lower 6 in. of the column.

After the initial NDE was completed on the settling column, the three 2.5-in.-diameter sample cylinders were removed and subsequently X-rayed. No evidence of liquefaction was found in the sample cylinders resulting from the transport to 306E. The sludge compacted slightly, but the layers remained distinctly defined (Figure 8). Apparently, “wall effects” in the small-diameter cylinders protected the layered sludge profile. The concentration of W/Co fragments in the upper 5% to 15% of the sample cylinders appeared to be reduced. In the lower 85% to 95% of the cylinders, the NDE showed uniform overall vertical distribution of W/Co fragments (Figure 9). On a sub-layer by sub-layer basis, some segregation of the stainless steel (gray areas) and W/Co fragments can be seen. It is presumed that an equivalent W/Co distribution existed in the well-defined layers in the 1-ft-diameter settling column before it was transported to 306E.

After the three sample cylinders were slowly removed, the settling column was again examined via X-ray. Even though the sludge was significantly disturbed when the sample cylinders were withdrawn, the distribution of the W/Co was largely unaffected (i.e., no significant segregation as the sludge and water moved to fill in the voids left behind when the cores were pulled).

In summary, the settling model proposed for the LDC sludge filling operation was clearly confirmed by the settling test. Because of the methods used and consistent behavior observed, no further settling model confirmation testing is proposed. While the settling test was not designed to simulate LDC transport nor

to evaluate the effects of liquefaction, the observed instability of the defined layers in the 1-ft-diameter settling column suggests that some liquefaction could occur in the 5-ft-diameter LDCs. Further evaluation of the liquefaction potential of the LDC should be considered.



Figures 5a and 5b. Settled Sludge Before Being Transported to NDE Facility (306E Building).
Figure 5a shows the sludge level in the sample cylinders is higher than that of the settling column. Figure 5b shows 20 distinct layers.



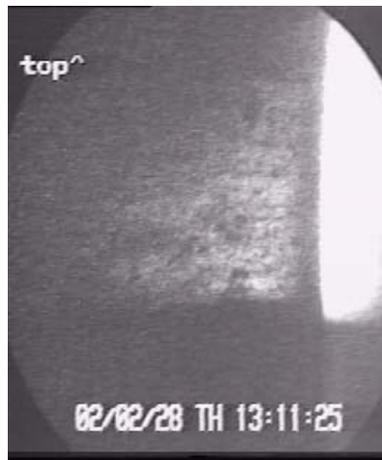
Figures 6a and 6b. Settled Sludge After Transport to NDE Facility (306E Building).
Only the lower four layers remain distinct; other layers have rotated or collapsed.



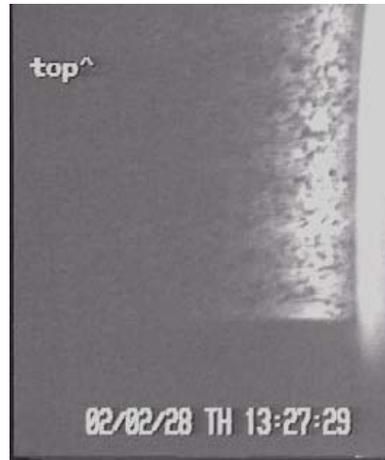
(a) 0 – 2 in. from top



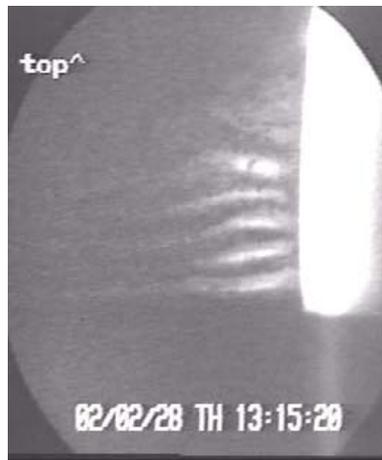
(b) 2 – 4 in. from top



(c) 4 – 6 in. from top



(d) 6 – 8 in. from top

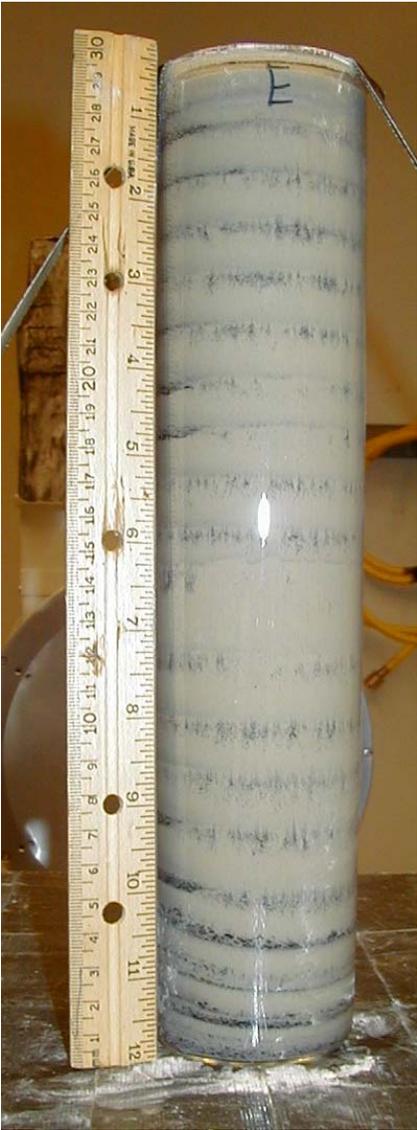


(e) 8 – 10 in. from top

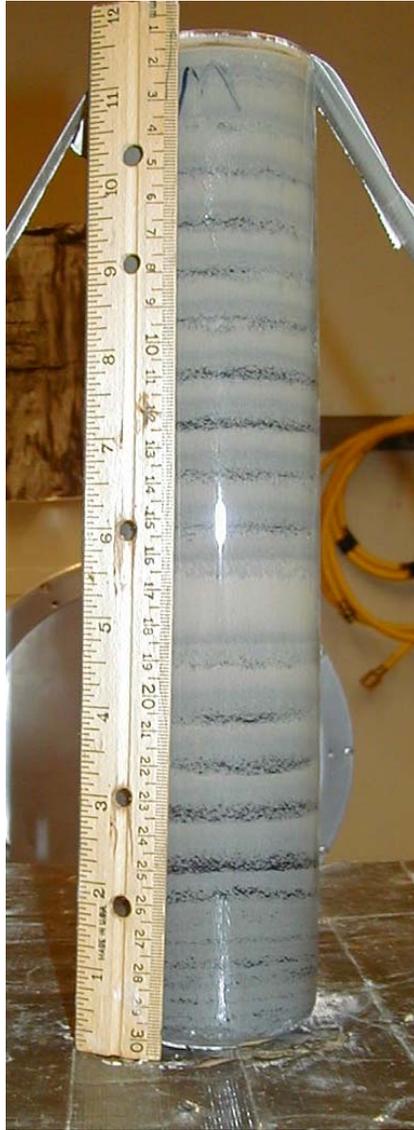


(f) 8 – 10 in. from top

Figure 7. X-ray Images of Settling Column After Transport to NDE Facility (306E Building). Black spots are W/Co fragments.



(a)



(b)

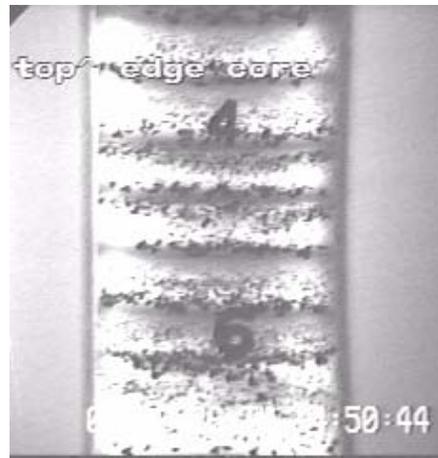


(c)

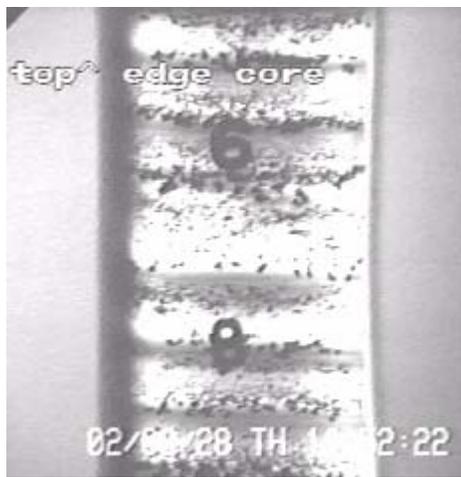
Figure 8. Sample Cylinders Pulled Out of Settling Column After Transport to NDE Facility (306E Building). Distinct layers are still evident. Samples are: (a) core from edge (next to wall) of column, (b) core from between the center and edge, and (c) core from center of column.



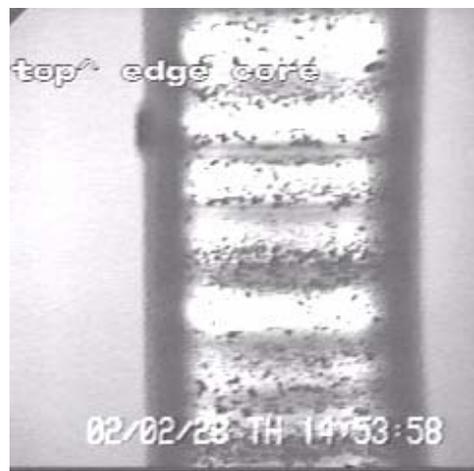
(a) 0 to 4 in. from Top



(b) 4 to 6 in. from Top



(c) 6 to 8 in. from Top



(d) 8 to 10 in. from Top



(e) 10 to 12 in. from Top (Bottom)

Figure 9. X-ray Images of W/Co Distribution (Black Spots) in Sample Cylinder Located Adjacent to Column Wall. Gray zones are areas rich in stainless steel powder.

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